

Chapter 4

Sampling and Analysis Protocols

4.1 General

This chapter provides guidance to USACE personnel and USACE contractors for using the sampling and analytical instructions in the appendices to this manual and for developing project-specific instructions if project-specific characteristics make it impractical to use the sampling and analytical instructions found in these appendices. Issues other than those identified in the general SAP format requirements found in Chapter 3 may have to be included in the SAP to meet project-specific regulatory requirements. To meet project-specific protocols and satisfy any additional requirements, additional field and analytical SOPs and references have been included in Appendix A. General guidance for developing additional site-specific instructions has been included in this chapter. With respect to sampling and analytical protocols, the information provided herein may be used to prepare the SOW for the project or to prepare the SAP. In some instances, data collection activities will occur that are not covered by this manual. The references in Appendix A and the discussion in paragraph 4.4 may be useful under these circumstances.

4.2 Selecting Sampling and Analytical Instructions

As discussed in paragraph 2.3, selection of sampling and analytical protocols for a specific site is dependent upon the site constraints, data needs and data quality objectives, and sampling strategies for the various media. After an analysis of these factors has been completed, sufficient information should exist to select appropriate sampling and analytical instructions from the appendices in this manual. As discussed in EM 200-1-2, sampling and analytical options and appropriate SOPs or instructions should be selected after consideration of the following criteria: schedule, regulatory, technical (effectiveness and implementability), and budget. Guidance to follow during the selection process is provided in the following subsections.

4.2.1 Sampling instructions. Information gathered from Steps 1 and 2 discussed in paragraph 2.3 should be used to identify applicable sampling protocols from the instructions in the appendices. An analysis of the constraints at the site will provide information needed to propose sampling locations and sampling procedures. This analysis should consider the media to be sampled, the types of contaminants, and the physical characteristics of the site. Project resource constraints will also be a factor. An analysis of data needs and DQOs will identify applicability of filtration, compositing, and homogenization procedures and field QC requirements. Sampling strategies should also be reviewed to determine the location and frequency of samples. After this information has been reviewed, appropriate sampling method options may be developed from the instructions in the appendices. If the instructions in the appendices do not contain an appropriate sampling method, alternative methods may be developed using the relevant references in Appendix A and the procedures described in paragraph 4.4.

4.2.2 Analytical instructions. The information needed to properly select an analytical instruction can be obtained from following Steps 1 and 2 of paragraph 2.3. Analysis of the constraints at the site provides information about the sample matrix, measurement parameters, and regulatory and customer preferences in regard to the type of analytical method to be used. A review of the data needs may define additional sample handling procedures, including homogenization and subsampling requirements, detection and quantitation limit requirements, instrumentation requirements, and appropriate analytical methods. After this information has been reviewed, appropriate analytical options may be identified from the instructions in Appendix I. If the instructions identified in Appendix I do not contain an

appropriate analytical method, the relevant references in Appendix A and the procedures in paragraph 4.4 may be used to develop additional instructions.

4.3 Additional Standard Operating Procedures

If the appendices do not contain appropriate sampling and analytical protocols, it will be necessary to develop additional instructions. Paragraphs 2.3 and 4.2 of this manual should be consulted when deciding if other instructions need to be developed. Paragraph 4.4 discusses the methodology for developing instructions. The references in Appendix A contain information that may be used to develop additional instructions.

4.4 Development of Project-Specific Protocols

As previously discussed, it may be necessary to develop sampling and analytical protocols other than those identified in the appendices. Additional instructions may be required for a myriad of reasons: client preferences, regulators' preferences, unusual site conditions, budget considerations, etc. If it is determined that new sampling and analytical protocols need to be developed, or protocols other than those found in the appendices are preferred, then this section and the references in Appendix A can be used to develop the new protocols. However, it is important that the new instructions are able to satisfy data needs and DQOs as well as satisfying scheduling, regulatory, technical, and budget criteria. Guidance for developing new instructions follows. Additional information can be found in several of the references in Appendix A, especially EPA/600/2-80/018, EPA 600/4-79/020, EPA SW-846, and EPA/540/P-87/001.

4.4.1 Sampling instructions. The following list is a template that may be used as an outline to develop new sampling instructions. The references in Appendix A provide additional guidance.

- (1) Scope and purpose.
- (2) Definitions.
- (3) Applicability.
- (4) Sample locations.
- (5) Applicable sampling strategies (discrete/composite: random, judgmental, stratified, etc.).
- (6) When filtration is applicable (sampling for dissolved metals).
- (7) When homogenization is applicable (sampling solid media).
- (8) Method specified in entirety (step-by-step presentation).
- (9) Field QC requirements (all field duplicates, all QA splits, trip blanks, background samples, highly contaminated media rinsates).
- (10) Split-sample techniques/deviations from normal protocol.
- (11) Preservation techniques (cool, acid preservation, base preservation, chlorine binding).

- (12) Field measurements.
- (13) Miscellaneous considerations.

4.4.2 Analytical instructions. The following list is a template that may be used as an outline to develop new analytical instructions. The references in Appendix A provide additional guidance.

- (1) Title/Signature/Effective Date page
- (2) Scope and application, including applicability to various matrices and discrete/composite procedures.
- (3) Method summary
- (4) Sample preservation
- (5) Containers, handling, and storage
- (6) Interferences and potential problems
- (7) Equipment and apparatus
- (8) Reagents and solutions
- (9) Procedures
 - Applicable sample preparation and cleanup procedures.
 - Any applicable special sample handling requirements.
 - Analytical method specified in entirety (step-by-step presentation).
 - Instrumentation requirements.
- (10) Calculations
- (11) QC requirements for second-column confirmation, and/or the analysis of surrogates, matrix spikes, internal standards, blanks, laboratory control samples. All QC elements should define appropriate measurement quality objectives (MQOs) for appropriate data quality indicators (i.e., precision and bias).
- (12) Corrective actions
- (13) Data evaluation
- (14) Method detection limit studies/sensitivity assessment
- (15) Analyst experience requirements

- (16) Health and safety
- (17) Sample disposal
- (18) References
- (19) Definitions
- (20) Example forms